IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:
Arjun G. Yodh et al.

Confirmation No.: **7568**

Application No.: **10/526,941**

Group Art Unit: **1793**

Filing Date: **September 8, 2005**

Examiner: **Brittany M. Martinez**

For: **Carbon Nanotubes: High Solids Dispersions and Nematic Gels Thereof**

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

DECLARATION PURSUANT TO 37 C.F.R. § 1.131

I, Mohammad F. Islam, hereby declare that:

1. I am an inventor of the invention described and claimed in U.S. Patent Application Number 10/526,941 (hereinafter referred to as "the 941 application"), filed September 8, 2005, in the United States Patent and Trademark Office.

2. I am aware that the pending claims of the 941 application have been rejected as being unpatentable over U.S. Patent Application Pub. No. 2003/0133865 ("Smalley"). It has been explained to me that Smalley was filed on July 2, 2002, but that it claims priority from four U.S. provisional applications (collectively referred to as "the Smalley provisionals"):

Serial No. 60/303,469, filed July 6, 2001;
Serial No. 60/303,470, filed July 6, 2001;
Serial No. 60/337,561, filed November 8, 2001; and
Serial No. 60/337,951, filed December 7, 2001.

3. It has been explained to me that none of the Smalley provisionals disclose the use of at least one surfactant comprising an aromatic group.

4. In accordance with CFR § 1.131, as an inventor of the subject matter of the pending claims, and without conceding the propriety of the rejections of the pending claims, I hereby declare that I invented the subject matter with the inclusion of at least one surfactant comprising an aromatic group prior to July 2, 2002. I further hereby declare that I worked diligently from a date prior to July 2, 2002, to the date of constructive reduction to practice, September 10, 2002, the priority date of the 941 application, in order to prepare the 941 application and patent the invention.

5. In support of the instant declaration, a copy of relevant pages of a laboratory notebook prepared during the development of the claimed invention is attached hereto (Attachment B), which was created prior to July 2, 2002. The date range is from May 22, 2002 through July 20, 2002, which provides evidence of conception of the invention prior to the effective date of the Smalley reference and evidence of due diligence from prior to said date to the filing date of the provisional application, September 10, 2002.

6. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information or belief are believed to be true; and further, that these statements were made with the knowledge that willful false statements and the like are punishable by fine or by imprisonment, or both, under § 1001 of Title 18 of the United States Code, and that such willful statements may jeopardize the validity of the application, any patent issuing thereupon, or any patent to which this verified statement is directed.

Date: 01/20/2010 Signature: M.F. Islam

Mohammad F. Islam

Exhibit A
Mohammad F. Islam

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Education

2000 Ph.D. Physics, Lehigh University, Bethlehem, PA 18015
1996 M.S. Physics, Lehigh University, Bethlehem, PA 18015
1994 B.S. Physics,

Work Experience

2005-Present Associate Professor, Chemical Engineering and Materials Science and Engineering
Carnegie Mellon University, Pittsburgh, PA 15213

2002-2005 Postdoctoral Fellow, Department of Physics and Astronomy
University of Pennsylvania, Philadelphia, PA

Honors and Awards

2007 Alfred P. Sloan Research Fellow
2007 National Science Foundation Career Award
2006 American Chemical Society PRF Award
1999 Sigma Xi
1997 Hoechst Celanese Award for Excellence in Polymer Science

Current Research Interests

Novel Self-Assembly and Phase Transformations in Single- and Multi-Component Systems: Formation of diverse structures as well as phase transformations in multi-component systems using temperature sensitive colloidal particles.

Utilizing Nanomaterials to Investigate Cellular Functions: Developing novel nanomaterial based vectors and investigating changes in cellular functions due to internalization of these vectors.

Carbon Nanotube Based Porous Materials for Energy Applications: Created ultra-light, highly porous materials with carbon nanotubes; Investigating use as electrodes and support for catalyst particles.

Dependence of Cell Functions on Substrate Properties: Developed polymeric hydrogels with tunable local stiffness and organization; Using materials to probe dependence of cellular functions on substrate stiffness and spatial organization.

Exhibit B

05/22/02

Make 2 samples of 1% Hipco + 10X NaDDBS in water
each sample weighs 3 g

add Hipco ~~0.03 g~~	0.0321 g	0.0324 g

Make

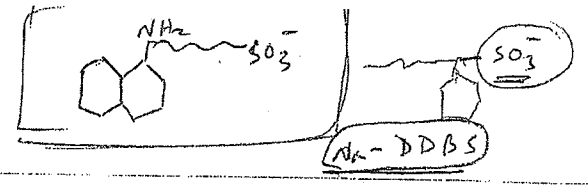
~~add~~	NaDDBS stock	2.002 g	19.96 %
	Total	10.03	

	Hipco	0.0321 g	0.0324 g
Add	NaDDBS	1.5221 g	1.5103 g
	water	1.4653 g	1.4762 g

Sample #3

Hipco	0.0314 g
NaDDBS	1.501 g
water	1.480 g

Dioxy Choalate



NT Project:

1. Scheme to separate & stabilize CNT without damaging it.

Tip Sonication (low f, high power) ~~Bath~~ Bath Sonication (high f)

- Advantage over pyrene functionalized CNT (damages CNT & destroys electronic property)

- comment on surfactant type for longer & better stability & separation
  * Benzene & charge surfactant best (NaDDBS)
  (Literature is mostly on SDS
  one paper ODA)

2. Fractionate CNT using either HPLC, SEC
   ↑ we use this
   or GPC
   ↑ Can use this

One paper on using GPC but do not elaborate or show careful results. (Dresberg's group)

- Can use this technique for other Rod shaped ~~xx~~ particles.

3. Characterization:

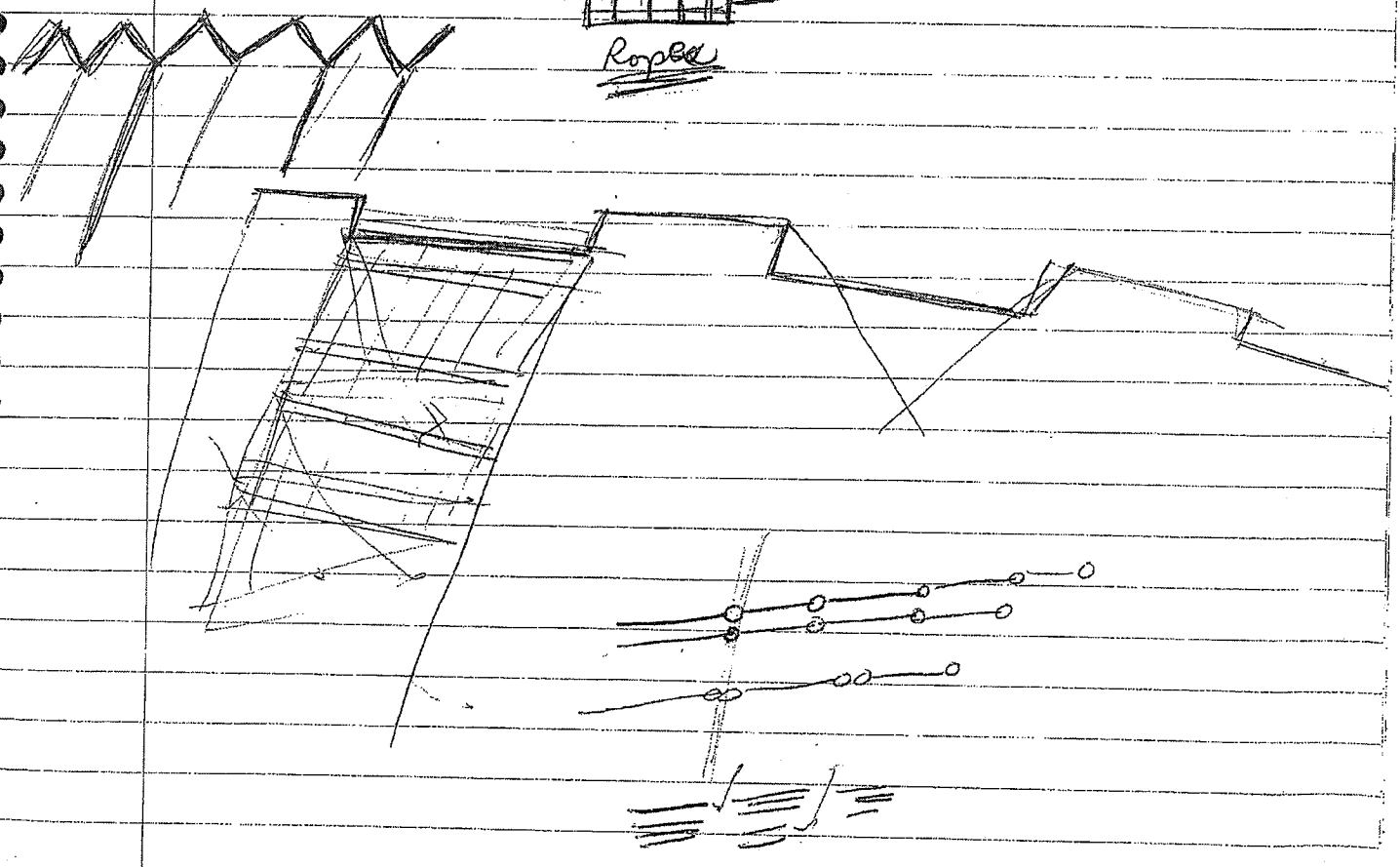
Use mainly LS ← large statistics

Cross check for a few structures w/ AFM

4. Impact: stable, single CNT dispersions

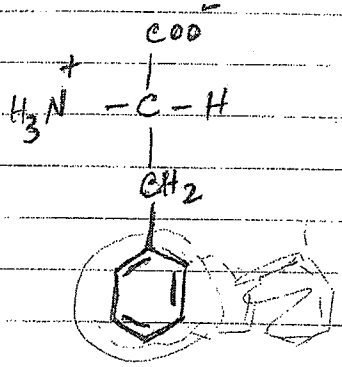
Assembly, Controlled deposition...

Ropes



05/19/02 Bill Degrado

more surfactants to try:

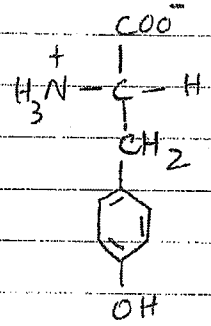


Phenyl alanine

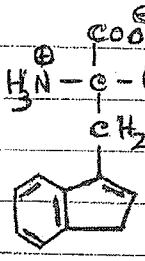
(F)

Phe

or Trimer of F (F3)



Tyrosine



Tryptophan (W)

[Trp]

Also try Chaps & PEG

chiral proteins Bill Degrado JMB 2001

Butyl group, OH, benzene, amine group

Can we make chiral molecules to wrap around!

Use Hydrogen bonding.

06/18/02

Current Status of NT dispersion & characterization:

1% NT in NaDDBS

Bath sonic dispersion

- electrophoresis through 0.5% Agarose gel
  - cut gel into small segments
    - ultra centrifuge gel through filter
      - most HiPco gets stuck in filter
      - melt gel at 95°C & then ultra centrifuge HiPco out
        - successful
          - need good technique
          - abandon
- Run through Sepharose 2B
  - sample comes through the gel, some of the solution gets trapped into the gel (long elution time)
  - They are presumably amorphous Carbon, buckyball etc
  - collect sample in multiple vials.
  - next step
    - look at a few of them to see how good is the separation
    - Try this approach more
- AFM of sample as is
  - unsuccessful; sample does not stick to surface
  - modify surface
    - saline
      - Similar as poly-L-L
      - check surface quality & see if NT sticks to the surface

tipsonic dispersion

- electrophoresis through 0.5% Agarose gel
  - most did not go through; seems tip is not a good technique to disperse HiPco
  - Abandon this method
- AFM of sample as is
  - unsuccessful; sample does not stick to surface
- modify surface
  - polylysine
    - successful; ~~soaked~~ HiPco sticks to surface after dipping into soln of HiPco
    - next step check the surface quality & see how NT looks like

07/01/2002

50 μl of sample placed on chip wait for 5 min & then shaken off

MI 10-4.000 Silane treated 10^{-4} HipCo 5μm Scan in NaDDB (Too much surf)

MI 10-6.001 " " 10^{-6} " " (Too much surfactant)

MI 10-6.002 diff area, same chip

→ 10-4.003 10^{-4} chip rinsed in H_2O ~2min

10-4.004 diff area, same chip

10-4.005 10^{-4} prepared by dunking in soln & then rinsing with water, dried with canned air

07/08/2002

Prepare 4g of 0.01% NT in NaDDBS From 1% NT in NaDDBS

1% NT soln	~~0.049~~ 0.0447g	0.01%
water	4.4434g	

07/08/02

AFM on NaDDBS stabilized HiPCO

ddbs10-4.000	200µl on the surface, leave it on for 10min, spin it at 3000 rpm then add 2ml of water as the sample is spinning. → no feature
ddbs10-4.001	same sample - diff location
ddbs10-4.002	dipped in soln, ~~then~~ for 5 min, then gently rinse it in a water bath - lots of NT
ddbs10-4.003	ZOOMED in some where in the previous ~~pic~~ picture
.004	different Place on sample (SP)
.005	Zoom in of .004 (SP)
.006	new location 10µ (SP)

ddds10-4.007

12th cut of gel phoresis sample

conc ~ 10^{-4} NT in NaDDS

I dipped the clasp in soln & then dried & then rinsed in water.

07/11/02

What I need to do:

conc	SDS	TX100	NaDDBS
Prepare 1% → dilute to 0.01%			
↑ Sonicate	↑ donot sonicate; just dilution		
Prepare 0.01% sample ✓✓			✓
↑ Sonicate			

I want to make 10% HiPCO soln,

9.96% → HiPCO 0.2000g

→ Add Rest 20% NaDDBS to get 2.0099g

→ Final wt 2.0061g

07/12/02

Make 20 wt% Tx-100 stock soln (50 grams)

Tx-100 add 10 g ~~#~~ 10.0061 g

Rest add water till 50g Total 50.0274 g

Make 4gpl % ~~Tx-100~~ NT soln w/ Tx-100 (10%)

NT = 0.0397 g
Tx-100 (20 wt%) = 2.0085 g
water = 1.9913
} ~1% (0.984)

Also make 10^{-4} soln from 10^{-2} soln for ~~AFM~~

SDS NT

1% soln = 0.0458 g
Add water till = 4.6047 g
} ~1×10^{-4} (6.9×10^{-5})

NaDDBS NT

1% soln = 0.0405 g
add water till = 4.0114 g
} ~1×10^{-4}

07/18/02

NT samples I have been looking at:

A

NaDDBS coated NT

10^{-4} dipped, rinsed

Baked

ddbs dep. 000 ← 5μm

ddBs 10-4.040

— .051

4μm scan

A1

Same as A but not baked

2μm scan

naddbsa. 026

— .037

naddbs 10-4.021

5μm scan ↓ .026

discard double tip

E1

1% NaDDBS NT spun at 6000 rpm while I pipetted on NT soln

before rigorous rinsing: ~~ddbs~~ Naddbse1.000 — .004

After rinsing: naddbse1.008 — .015

Same baked

naddse1.040

bad

B2 NaDDBS NT

1% 4000 rpm

~~NaDD~~ soln dropped on chip was spinning

naddbsb2.000 — .002 } before cleaning

naddbsb2.003 ← After cleaning

Baked

naddbsb2.040 — .042 } same way prepared sample

overlapping Conc. NT

E

NaDDBS 1%

(Sample 1 month old)

Spins deposited

naddbse.000 ← before clean

.001 ← After clean

— .007 } conc. NT

Baked

Naddbse.040 — .042 } overlapping conc. NT

B

NaDDBS NT

1%

6000 rpm spin deposited 2μ(

Naddbsb.000 — .001 } I merge confusion

Baked

28% 70/307

158/307 ~ 60%

212/307 ~ 73%

C

10^{-4} from 10^{-2} dipper

Naddbsc.000 } before baking

naddbsc.040 } after baking
.048

no tube

F

10^{-4} from 10^{-2} dipper sample(1)

After baking

naddbsf.040 } no tube
.041

Cut 12 new 12-2

ddbs 12n.000 } Long tubes
.005 }

ddbs 12-2.000 } not tubes

12nd cut New-2

4th cut New1

ddBS 4N1,000
↓
,009

Found single tubes

20th cut

27th cut

C2

dirty not tubed

10^{-4} from 10^{-2} dipped, Rinsed & Baked

C3

10^{-4} from 10^{-2} dipped, Rinsed baked

a lot of tubes

F2

10^{-4} from 10^{-2} (sample ①) dipped, Rinsed baked

Naddbs F2,000
↓
,009

G1

10^{-3} from 10^{-4}

dry & UV, rinsed & baked

C4

10^{-4} from 10^{-2} dipped, rinsed, baked

a lot of tubes but chip is dirty

H1

Bake surface Silane treated, Rinsed baked

H2

same as H1 but not Rinsed & Baked

07/20/02

Prepare high conc. of Laser Tuber

Take 20 mg of 5×10^{-3} % wt tuber in a glass bottle & slowly evaporate water to increase conc.

Empty bottle w/o cap = 13.9254 g

w/ tube w/o cap 28.9276 g

∴ tube wt = 15.002 g at 0.5% wt

slow evaporate at 44°C for several days

After some solvent evaporation w/ tube w/o cap = 17.4744

∴ tube soln wt = 3.549

New conc.

New conc = 2.113×10^{-2} by wt = 2.113 % by wt

07/24/02

Prepare 0.1% NaDDBS-NT soln to sonicate

1% NaDDBS-NT = 0.4038g
Add water till = 4.0127g
} ≈ 0.1%

Prepare 0.1% SDS-NT soln

1% SDS-NT = 0.4009g
Add water till = 4.0193g